NASA/CR- 1998 208238

NAG-1-1814

IN-38-CR

Technologies for Nondestructive Evaluation of Surfaces and Thin Coatings

Final Report

1 Introduction and Overview

The effort included in this project included several related activities encompassing basic understanding, technological development, customer identification and commercial transfer of several methodologies for nondestructive evaluation of surfaces and thin surface coatings. Consistent with the academic environment, students were involved in the effort working with established investigators to further their training, provide a nucleus of experienced practitioners in the new technologies during their industrial introduction, and utilize their talents for project goals.

As will be seen in various portions of the report, some of the effort has led to commercialization. This process has spawned other efforts related to this project which are supported from outside sources. These activities are occupying the efforts of some of the people who were previously supported within this grant and its predecessors.

The most advanced of the supported technologies is thermography, for which the previous joint efforts of the investigators and NASA researchers have developed several techniques for extending the utility of straight thermographic inspection by producing methods of interpretation and analysis accessible to automatic image processing with computer data analysis. The effort reported for this technology has been to introduce the techniques to new user communities, who are then be able to add to the effective uses of existing products with only slight development work. In a related development, analysis of a thermal measurement situation in past efforts led to a new insight into the behavior of simple temperature probes. This insight, previously reported to the narrow community in which the particular measurement was made, was reported to the community of generic temperature measurement experts this year. In addition to the propagation of mature thermographic techniques, the development of a thermoelastic imaging system has been an important related development. Part of the work carried out in the effort reported here has been to prepare reports introducing the newly commercially available thermoelastic measurements to the appropriate user communities. This presentation represents culmination of an effort which was initiated under a predecessor of this grant long before the measurement was commercially available. The availability of the full-field synchronous analysis of thermal data has opened opportunities for synchronous techniques which can extend the sensitivity of thermographic measurements by imposing suitably configured periodic heating stimuli. One such technique has been used in a related "daughter" project for measurement of thickness of a thin coating, such as paint. Another application, again now part of a separate project, involves using the full-field synchronous demodulation technique in visible light to obtain images of photoelastic responses at sensitivities far below a single fringe. At present, the College of William and Mary is the only organization outside of NASA which has this capability operational in visible light. The separate project, discussed below as related work, has the ambitious goal of developing an industrially useful full-field stress separation from a combination of thermoelasticity and photoelasticity.

The effort in Optically Stimulated Electron Emission (OSEE) this year has been directed primarily towards producing a hand-held instrument for potential commercial use, and it has been directed and executed entirely within the engineering development activity in the Nondestructive Evaluation Science Branch of NASA Langley Research Center. Efforts under this grant have related to this project in an advisory capacity. Other efforts, discussed below, have related to education and the identification of possible users of the technology. As part of this effort, the laboratory OSEE system has been relocated to the College of William and Mary and placed in operation there.

The effort in scintillator analysis, to characterize the absorption and luminescence characteristics of glass fibers doped with terbium, is expected to come to a conclusion during the current grant period. A presentation on the results has been given at a national scientific meeting.

2 Activities and Accomplishments

The work described in this section, while addressing the entire project, highlights the final year of the effort. Previous reports, submitted on an annual basis, have covered some aspects of the work more extensively.

2.1 Combined thermoelasticity and photoelasticity

The efforts of one of the two supported graduate students this year have been directed towards issues of coating development for combined thermoelasticity and photoelasticity. To review this effort, which is the heart of a related STTR, the objective is to develop a method of determining full-field measurements of the stress tensor on the surface of an in-service structure. The determination is done by combining two existing methods of stress analysis: reflection photoelasticity and thermoelasticity. Each of these methods responds to stress (as indicated by strain) on the surface of a structure, but neither is complete by itself. The missing information associated with each technique is provided by the other, so the two measurement methods are, in principal, complementary. Each of the methods depends on a coating: a birefringent coating for photoelasticity and an emissivity-enhancing coating for thermoelasticity. The critical factor in combining the methods into an NDE technique is to formulate a coating which is suitable for both methods. If the coating furthermore can act as a protective coating, it can serve as the service–protection coating for the structure as well as the diagnostic NDE coating. The effort being undertaken within this project has been associated with a search for an optimum coating and determination of an appropriate coating application method.

Most commercial photoelastic coatings are opaque and highly absorbing (as contrasted with reflecting) in the infrared band from 8-12 microns in wavelength. The most effective thermoelastic measurement system uses array technology with parallel processing, and infrared sensing arrays are generally available with sensitivity in the 3-5 micron band. Many polymeric materials which absorb in the 8-12 micron band are less absorbing or even transparent in the 3-5 micron band. For one effective approach to producing a single dual-use coating, the coating must be transparent to visible light and opaque to infrared radiation. Thus, the task being undertaken is a dual one: to find an appropriate polymer and to produce a convenient and effective method of applying it to the surface under test. To a large extent, the success achieved in developing the coating will determine the eventual marketability of the resulting technology. This factor has been considered important enough that, along with the availability of a graduate student at the appropriate stage of her academic career (Ms. Johnson), the effort in this direction has been considered appropriate for pursuit under this grant.

Along with application techniques, a variety of polymers have been examined as preliminary choices for a possible thermoelastic-photoelastic coating. These have been found with the aid Dr. Catherine Fay, a National Research Council Postdoctoral Fellow at the Polymers Branch of NASA Langley Research Center and Professor Floyd Klavetter, of the Applied Science Department of William and Mary. Besides the commercially available photoelastic coating, a Bisphenol-A based epoxy resin, films received for evaluation include, by common name, LaRC-1A, Kapton HA, UPILEX R, LaRC 8515, TOR, Kapton HY, teflon, mylar, Upilex 5 and LaRC-1Ax. Each of these materials exhibits some birefringence. The samples varied in color from clear to a brownish-orange, in visible light optical transmission from transparent to opaque, and in thickness from 25 to 75 microns. Because of their chemical structure, the materials were expected to be opaque in the infrared, but tests in the 3-5 micron band showed them to be generally transparent. This transparency should be able to be alleviated with the addition of different chemicals to the formulation. From qualitative initial tests, Kapton HA, TOR and Upilex 5 showed the greatest birefringence of the ten films when stressed. The commercial photoelastic coating seems to be both opaque in the 3-5 micron band but also has substantial birefringence. Thus, it seems to be the best of the candidates tested. In phase 2, further searches will be made for coatings, and quantitative determinations of the strain-optic coefficient will be developed and applied.

Parallel work on the combination of thermoelastic and photoelastic stress analysis has been ongoing in England, at two universities, The University of Sheffield, under the direction of Dr Eann Patterson and the University of Liverpool, under the direction of Dr. Janice Dulieu-Smith. The Applied Science Department at William and Mary was pleased to attract Dr. Dulieu-Smith for a seminar, at which she presented several of her thoughts in exchange for a demonstration of the thermoelastic capability at the College. In addition, we were able to share information with collaborators and recent graduates of Dr Patterson's research team at a meeting of the Society of Experimental Mechanics in Nashville.

2.2 Thermoelasticity

Acceptance of a new technique in the industrial sector frequently requires a concerted, long-term effort in communication as well as simply innovation. This requirement seems to apply to thermoelastic stress analysis, and one of the major impediments to industrial application has been the time required to make a measurement. As rapid measurement is one of the hallmarks of the Stress Photonics DeltaTherm 1000 system, developed under SBIR support in conjunction with NESB at LaRC, one method of communicating and establishing credibility for the technique was begun in November 1993 by participation in a workshop in Ft. Worth, Texas, entitled "Nontraditional Methods of Sensing Damage in Materials and Structures," under the auspices of the American Society of Testing and Materials (ASTM). In part because of the success of this presentation, an invitation was received to participate in a symposium on May 20 of this year in Orlando, Florida, entitled Symposium on Nontraditional Methods of Sensing Stress, Strain, and Damage in Materials and Structures. One paper was prepared and another collaborated in: "An Array Measurement System for Thermoelastic Stress Analysis," and "Stress Intensity Measurement via Infrared Focal Plane Array," Both of these presentations were chosen to be considered, following peer review, for inclusion in a Special Technical Publication (STP) of ASTM. The two manuscripts are appended to this report, and they are both in the final stage of editorial review following peer acceptance. The inclusion of these two papers in a publication on ASTM is expected to support the credibility of TSA as an industrially accepted "new technique" and, in the process, provide well-deserved recognition to the NASA-supported efforts to develop this NDE measurement tool.

2.3 Thermography/Temperature Measurement

In previous years, a temperature measurement analysis was done for a situation in which a thermocouple probe was moved to a variety of positions for the purpose of obtaining a temperature profile. In particular, the temperature profile was used as a method of locating a particular feature observed on the profile - that is, as a position marker. Previously, in conjunction with another research group within NASA Langley Research Center the the Air Force Liaison Officer, a discrepancy was observed between such a marker and a radiographic position measurement. This discrepancy occurred in a Bridgman furnace used for crystal growth, and its cause and resolution were reported in the appropriate literature. The resolution involved a conduction correction to position in temperature sensors mounted on probes, a situation which can reasonably affect measurement in many instances outside of crystal growth furnaces. In order to reach this audience effectively, a presentation was made at the 42nd International Instrumentation Symposium of the Instrument Society of America entitled "Displacement Compensation of Temperature Probe Data." The presentation is available in reprint form from the proceedings of the symposium. A preprint is attached as an appendix.

An interesting application of infrared thermography was presented to the Applied Science Department by the Colonial Williamsburg Foundation (CWF) regarding industrial hazard evaluation. Each year in the autumn, CWF constructs a colonial era brick kiln to fire the bricks which have been made during the year by historically accurate interpreters of the building trades and visitors under their guidance. Operation of the brick kiln and other facilities in the historic recreation must conform to modern standards of safety, a departure from strict historical accuracy which is strongly supported by the Foundation and its visitors. As part of the continual search for safety verification, the safety officer for CWF asked for an independent evaluation of the radiation hazard to employees and visitors from operation of the kiln, which involves exposure to the mouths of the wood-fired furnaces which are built into the kiln for a period of about 1 week. While theoretical indications and intuitive opinions from several experts in radiation have uniformly been that no hazard exists, a measurement program was undertaken to determine the heat flux from the mouths of the furnaces as a double-check the indications.

2.4 OSEE

An activity of several years with the National Center for Manufacturing Science (NMCS) was brought to a close with a final report entitled, "Investigation of the Use of Optically Stimulated Electron Emission (OSEE) to Measure Contamination Levels on Printed Circuit Boards." This report marked the first attempt started to investigate the use of OSEE for an industrial process in cooperation with representatives of the potential user industry. It indicated clearly that some of the industry-supplied substrate/contaminant pairs were easily detectable using OSEE while others were not. The project has become a prototype for similar projects in its employment of user-supplied samples and the production of points on a dose-response curve as tools in determination of applicability. The final report is included as an appendix.

A major project within the engineering development group in NESB at NASA LaRC over the period of this project has been development of a hand-held one inch footprint OSEE sensor suitable for application in an industrial setting, sometimes referred to as "on the shop floor." In the proposal for the present effort, it was presumed that this sensor would be completed and delivered prior to the start to the grant and that the only requirement would be to provide consultation on the application of the instrument. As it turned out, the instrument was not ready for demonstration until June of the grant year, and the consultation included some initial laboratory testing and preparation of a demonstration sample for cleanliness, as demonstrated with high OSEE readings corroborated with surface appearance. Further work was done with the engineering team to define the flow of argon into the lamp and measurement regions in order to accomplish a consistent reading while still conserving argon. Following a successful demonstration of the instrument in a video conference with users and supporters at NASA Marshall Space Flight Center and Thiokol Corporation, it was discovered that the lamp had little long-term stability and a short service life. These problems have moved the delivery date up, and consultation with the engineering staff continues. At the time of this report, the development of this instrument is still in its finishing stages. The woking prototype instrument is anticapated in a matter of weeks.

During the year and following the completion of all of the work required for the project with NCMS, the (old) laboratory apparatus for performing OSEE measurements was transferred by loan to the College of William and Mary, where it has been put back into operation. It is being used in conjunction with some College-supported research into OSEE response in increasing vacuum. A summer (Research Opportunities for Undergraduates) investigator, Ryan MacAllister, learned about OSEE and performed some studies of the effect of exposure to ambient air on stainless steel samples. This determination was in support of the design of sample handling apparatus. He also helped in the design of an OSEE instrument capable of performing measurements in a vacuum. A rising senior, Bon Woo Lee, is also using the apparatus in his Senior Research Project, which includes obtaining the first vacuum measurements for the OSEE device and, in the process, bringing a new vacuum system into operation.

2.5 Scintillator Characterization

The supported student (M. West) in this activity presented a paper with his principal research results to the American Physical Society March Meeting in St Louis, Missouri. The paper was entitled "Time evolution of radiation-induced luminescence in terbium-doped silicate glass". An abstract of the presentation is included in the appendix. Mr. West successfully defended his dissertation in 1997.

2.6 References and papers presented

Johnson, D. F., D. B. Opie, H. E. Schone, M. T. Langan and J. C. Stevens, "High–Temperature Superconduction Magnetic Shields Formed by Deep Drawing," *IEEE Trans. on Applied Superconductivity* 6(1), pp. 50-54, March, 1996 (reprint attached)

Lesniak, J. R., D. J. Bazile, B. R. Boyce, M. J. Zickel, K. E. Cramer and C. S. Welch, "Stress Intensity Measurement via Infrared Focal Plane Array," *Nontraditional Methods of Sensing Stress, Strain, and Damage in Maerials and Structures, ASTM STP 1318*, George F. Lucas and David A. Stubbs, Eds., 208-220, American Society for Testing and Materials, 1997.

Welch, Christopher S., James A. Hubert and Patrick G. Barber, "Displacement Compensation of Terperature Probe Data," *Proceedings of the 42nd International Instrumentation Symposium, Instrument Society of America,* May 5-9, 1996, San Diego, CA., pp. 225-234. (preprint attached)

Welch, C. S., Cramer, K. E., Lesniak, J. R., and Boyce, B. R. "An Array Measurement System for Thermoelastic Stress Analysis," *Nontraditional Methods of Sensing Stress, Strain, and Damage in Maerials and Structures, ASTM STP 1318,* George F. Lucas and David A. Stubbs, Eds., 198-207, American Society for Testing and Materials, 1997.

West, Michael S. and William P. Winfree, "Time Evolution of Radiation-Induced Luminescence in Terbium-Doped Silicate Glass," presented at the March Meeting of the American Physical Society, St. Louis, MO, March, 1996. (abstract attached)

3 Related Work

The work covered in this report was one of several related efforts which were mutually supportive in the Applied Science Department at the College of William and Mary. To illustrate the synergy, a brief description of the other efforts is included below.

3.1 Combined photoelastic and thermoelastic measurements

During the year roughly equivalent to this grant, the College of William and Mary was associated with a commercial firm, Stress Photonics, Inc., of Madison, Wisconsin, in Phase I of an STTR entitled "A Stress Imager Integrating Thermoelastic and Photoelastic Stress Analysis." The work at the College of William and Mary was directed towards the development of a dual-purpose easily applied coating material which could be used both for reflection photoelastic measurements and for thermoelastic measurements. As part of this work, some temperature-based methods were shown to be sensitive to thickness variations of the coatings in the range of interest Also shown was a sensitivity to photoelastic strain variations at sub-fringe levels, paving the way for thin photoelastic coating applications and delicate measurements. A Phase II proposal was written jointly by the collaborators, and it has been announced as selected, so when arrangements have been made, the work is expected to continue for the next two years.

3.2 Proposal to NSF for OSEE high-sensitivity process study

In the proposal for the work included in this report, it was noted that a proposal was under consideration at NSF to support a program examining in detail the process which causes OSEE to be very sensitive to some contaminants at very high sensitivity when the contamination amount is very small. This process has been hypothesized to be related to the change of work function on a contaminated surface, and changes in work function with contamination have been demonstrated for particular cases. However, it seems that experiments of the requisite delicacy to investigate the processes directly require a correlation with high vacuum surface inspection techniques, such as ultraviolet photoelectron spectroscopy, and support for the equipment required for such an undertaking has not been developed within the existing community of OSEE researchers and sponsors. The NSF review was completed during the project year, and the proposed effort was turned down, in part because of the size of the budget request. In turning the proposal down, some of the anonymous peer reviewers commented that the proposed research area was interesting and potentially important. A letter of support was also obtained from an interested industrial potential user of OSEE measurements. In view of the interest shown in the review of the project, the effort to characterize the high-sensitivity OSEE process is continuing, and appropriate sponsorship continues to be sought.

3.3 OSEE demonstration and evaluation

The support of OSEE technology has included efforts to broaden the base of users in order to develop a commercial market large enough to support a viable and responsive producer. The most effective technique for gaining access to industrial potential users has been demonstration projects modeled on the NCMS project originally undertaken by NASA. A project of this nature has recently been established between the College of William and Mary and Edison Welding Institute, of Columbus, Ohio. Inquiries have also been made in conversations with the president of Photoemission Technology, holders of the fundamental patent for OSEE as an NDE tool, and a verbal agreement has been made in principle to license the patent rights on a case-by-case basis for a nominal fee for activities in which OSEE technology is being demonstrated to or evaluated for potential new users.

3.4 Space Grant Fellowship

D. Johnson successfully competed for a Space Grant Graduate Fellowship during the year of this grant. Her research proposal was based on the combination of photoelasticity and thermoelasticity, and it clearly was associated with the effort under this grant. As part of the fellowship, she took part in a video presentation prepared by the Virginia Space Grant Consortium and Old Dominion University. This presentation, entitled Journey into Cyberspace, is aimed at secondary school students of both genders to interest them in science as a career. She also produced and gave a demonstration of combined thermoelasticity and photoelasticity in conjunction with a reception for the Virginia Space Grant Consortium.

3.5 Thermographic issues demonstration and development

A thermographic and thermoelastic apparatus has been loaned to the Applied Science Department of the College of William and Mary, and it is being used for teaching and research purposes. Thermography is routinely introduced in a laboratory setting to the students taking the general NDE class. It is also being used for outreach and demonstrations to classes in primary and secondary schools in the area. Continuing a practice initiated in former years, D. Johnson has introduced students to infrared thermography in a middle school of Henrico County,

4. Summary

The work carried out under this grant has carried forward research in several efforts related to nondestructive evaluation of surfaces and thin coatings. These have been generally related to the disciplines of optically stimulated electron emission (OSEE) and combined thermoelastic and photoelastic stress analysis as a tool in nondestructive evaluation. A by-product has been a deeper understanding of the operation of common thermocouple and thermistor probes. Associated work has been done in understanding X-Ray scintillator materials and magnetic shield fabrication and evaluation using high-temperature superconducting materials. The work has been mutually supporting with other work, and a related development of commercial technology is underway in two areas.